भारतीय मानक Indian Standard

इलेक्ट्रोप्लेटिंग के लिए इंडियम सल्फेट — विशिष्टि

IS 8590: 2023

(पहला पुनरीक्षण)

Indium Sulphate for Electroplating — Specification

(First Revision)

ICS 25.220.40

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भारतीय मानक ब्यूरो BUREAU OF INDIAN STANDARDS

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

Indium is plated on a base metal with a view to providing surface protection, particularly for inhibiting corrosion of cadmium and lead types of bearing alloys. The four most commonly used baths for electroplating indium are of cyanide, sulphate, fluoborate and sulphamate types. Indium sulphate is used in the preparation of sulphate electrolytes and is also supplied in the form of concentrated solution.

The Committee felt a need to revise the standard with a view to update the standard based on the experience of last four decades and on the currently available data.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

INDIUM SULPHATE FOR ELECTROPLATING — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for indium sulphate for electroplating.

2 REFERENCE

The Indian Standards given below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No. Title

IS 1070: 2023 Reagent grade water —
Specification (fourth revision)

IS No. Title

IS 4905: 2015 Random sampling and randomization procedures

(first revision)

3 REQUIREMENTS

3.1 Description

Indium sulphate shall be in the form of a white crystalline, deliquescent solid, readily soluble in water, free from foreign matter and visible impurities and shall correspond essentially to the formula $In_2(SO_4)_3.9H_2O$.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in co1 (4) of the Table.

Table 1 Requirements for Indium Sulphate

(Clause 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Solubility in water	To pass the test	A-2
ii)	Indium (as In), percent by mass, Min	33.75	A-3
iii)	Sulphate (as SO ₄), percent by mass, Max	42.38	A-4

4 HANDLING

Because of its highly hygroscopic nature, indium sulphate should be kept in well-closed bottles. While opening a bottle adequate care should be taken to prevent foreign particles from falling into the material and the closure should be replaced soon after withdrawing the material.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in air-tight containers, preferably with replaceable closure.

5.2 Marking

The containers shall be marked with the following:

- a) Name of the material;
- b) Net mass, with mass of equivalent indium (in bracket);
- c) Name of the manufacturer or his recognized trade-mark, if any; and
- d) Date and batch number of manufacture to enable the material to be traced from records.

5.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the

products may be marked with the Standard Mark.

6 SAMPLING

The method of preparing representative samples of the material and the criteria for its conformity to this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 3.2)

METHODS OF TEST FOR INDIUM SULPHATE FOR ELECTROPLATING

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 TEST FOR SOLUBILITY IN WATER

A-2.1 Procedure

Weigh accurately about 0.5 g of the material and dissolve in 50 ml of water. The material shall be taken as having satisfied the requirement of the test if there is no turbidity. Reserve the solution for further tests.

A-3 DETERMINATION OF INDIUM

A-3.1 General

Three methods of determination of indium have been given. These are based on: (a) precipitation of indium as indium hydroxide and ignition to indium trioxide, (b) precipitation with 8-hydroxyquinoline, and (c) titrimetric determination using EDTA. In the case of a dispute Method A shall be the referee method.

A-3.2 Method A — Precipitation with Ammonium Hydroxide

A-3.2.1 Reagents

A-3.2.1.1 *Ammonium hydroxide* — 10 N approximately

A-3.2.1.2 *Ammonium nitrate solution* — 2 percent (m/v)

A-3.2.1.3 *Dilute nitric acid* — 1:10 (v/v)

A-3.2.2 Procedure

Transfer the solution reserved in **A-2.1** to a 500 ml volumetric flasks and dilute to mark with water.

A-3.2.2.1 Pipette out 200 ml of the above solution into a beaker and heat to boiling. Add ammonium hydroxide until just ammoniacal and boil for 2 minutes. Cool to about 50 °C to 60 °C and stir in a little ashless filter paper pulp.

NOTES

1 Excess of ammonium hydroxide should be avoided as it may result in low recoveries of indium by peptizing the hydroxide precipitate. The filter paper pulp assists in collecting dispersed indium and in addition, is an aid to filtration.

2 If the solution from which indium is precipitated contains chlorides, especially ammonium chloride, the hydroxide precipitate should be dissolved in dilute nitric acid and reprecipitated and the fresh precipitate washed completely free from chloride, as indium is appreciably volatile in the presence of chloride during ignition.

A-3.2.2. Transfer the filter paper together with the precipitate to a tared platinum crucible. Carefully dry the contents, char the paper at as low a heat as possible, burn off the carbon and finally ignite at 750 °C to 800 °C under oxidizing conditions to constant mass.

NOTE — Indium trioxide is readily reduced at high temperature; hence it is necessary to burn off the carbon at as a low heat as possible under good oxidizing conditions.

A-3.2.3 Calculation

Indium (In), percent by mass =
$$\frac{82.70 M_1}{M_2}$$

where

 $M_1 = \text{mass}$, in g, of the residue; and

 M_2 = mass, in g, of the material in the aliquot taken for the test (**A-3.2.2.1**).

A-3.3 Method B — Precipitation with 8-Hydroxyquinoline

A-3.3.1 Reagents

A-3.3.1.1 Sodium acetate

A-3.3.1.2 Glacial acetic acid

A-3.3.1.3 8-Hydroxyquinoline solution

Mix 6 g of 8-hydroxyquinoline with 6 g of glacial acetic acid, dissolve in 150 ml of hot water and add ammonia until there is turbidity. Dilute to 200 ml, cool and filter.

A-3.3.2 Procedure

Pipette out 200 ml of the solution prepared in A-3.2.2 into a beaker. Add 2 g of sodium acetate and 2 ml of glacial acetic acid. Heat to 70 °C to 80 °C and add dropwise with stirring 8-hydroxyquinoline solution till precipitation is complete. Cool, stirring from time to time and allow to stand for 2 hours to 3 hours or for 10 minutes at 40 °C. Filter through a tared sintered glass crucible (G No. 4) removing a large part of the precipitant by transferring the precipitate to filter crucible with a small quantity of hot water and then with cold water (25 ml to 75 ml) until the filtrate is free from the yellow colouration. Dry the precipitate in the filter crucible for 1 hour to 1.5 hours at 120 °C. Cool and weigh to constant mass.

A-3.3.3 Calculation

Indium (In), percent by mass =
$$\frac{20.99 M_1}{M_2}$$

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where

 $M_1 = \text{mass}$, in g, of the residue; and

 M_2 = mass, in g, of the material in the aliquot taken for the test.

A-3.4 Method \mathbf{C} — Titrimetric Determination Using EDTA

A-3.4.1 Reagents

A-3.4.1.1 Standard EDTA disodium salt solution — 0.01 M

A-3.4.1.2 *Sodium potassium tartrate solution* — 5 percent (m/v)

A-3.4.1.3 *Buffer solution* — *p*H 8 to *p*H 10

Dissolve 13.4 g of ammonium chloride in water. Transfer to a 250 ml volumetric flask, add 88 ml of ammonium hydroxide (R.D. 0.90) and dilute with water up to the mark.

A-3.4.1.4 Indicator

Mix eriochrome black T and sodium chloride in the ratio 1:400.

A-3.4.2 Procedure

Pipette out 25 ml of the solution prepared in A-3.2.2 into a beaker. Add 25 ml of sodium potassium tartrate solution followed by 3 ml to 5 ml of buffer solution until the pH is between 8 and 10. Add indicator until a bright rose colouration appears. Heat to boiling and titrate with standard EDTA solution until the solution turns blue. At the end point heat again to boiling and wait for 30 seconds; the colour should be stable.

A-3.4.3 Calculation

Indium (In), percent by mass =
$$\frac{11.48 \text{ V} \times \text{M}}{M_2}$$

where

V= volume, in ml, of EDTA solution consumed;

M = molarity of EDTA solution; and

 M_2 = mass, in g, of the material in the aliquot taken for the test (**A-3.4.2**).

A-4 DETERMINATION OF SULPHATE

A-4.1 General

Sulphate is determined gravimetrically as barium sulphate.

A-4.2 Reagents

A-4.2.1 *Dilute Hydrochloric Acid* — approximately 5 N

A-4.2.2 *Barium Chloride Solution* — 10 percent (m/v)

A-4.3 Procedure

Pipette out 200 ml of the solution prepared in A-3.2.2 into a beaker. Add 25 ml of dilute hydrochloric acid. Heat to boiling, add 20 ml of hot barium chloride solution with stirring and allow to stand overnight. Filter through a tared sintered glass crucible (G No. 4), wash with water and carefully dry at 105 °C. Cool and weigh to constant mass.

A-4.4 Calculation

Sulphate (as SO₄), percent by mass =
$$\frac{41.15 M_1}{M_2}$$

where

 $M_1 = \text{mass}$, in g, of the residue; and

 M_2 = mass, in g, of the material in the aliquot taken for the test (**A-4.3**).

ANNEX B

(Clause 6)

SAMPLING OF INDIUM SULPHATE AND CRITERIA FOR CONFORMITY

B-1 GENERAL REQUIREMENTS OF SAMPLING

- **B-1.1** In drawing, preparing, storing and handling test samples, precautions given in **3** shall be strictly observed.
- **B-1.2** The sampling implements and the sample containers shall be clean and dry.

B-2 SCALE OF SAMPLING

- **B-2.1** In a single consignment all the containers from the same batch of manufacture shall be grouped together to form a lot.
- **B-2.2** For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected from the lot shall depend on the size (N) of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling

(Clause B-2.2)

Sl No.	Lot Size	Sample Size	
	N	n	
(1)	(2)	(3)	
i)	Up to 10	1	
ii)	11 to 50	2	
iii)	51 and above	3	

B-2.3 The containers shall be selected at random. In order to ensure randomness of selection, a random number table shall be used. For guidance and use of random number Tables, IS 4905 may be referred. In

the absence of random number table, the following procedure may be used:

Starting from any container in the lot, count them as 1, 2, 3,....., etc, up to r and so on, where r is the integral part of N/n (N being the number of containers in the lot and n the sample size). Every rth container thus counted shall be withdrawn so as to constitute the required sample size.

B-3 PREPARATION OF TEST SAMPLES AND NUMBER OF TESTS

- **B-3.1** From each of the selected containers 6 g of indium sulphate shall be withdrawn. These portions shall be thoroughly mixed to form a composite sample.
- **B-3.1.1** The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for use as the referee sample. These parts of the composite sample shall be transferred to suitable sample containers and labelled with full particulars for identification.
- **B-3.1.2** The referee test sample shall bear the seal of the purchaser as well as the supplier. It shall be kept at a place as agreed to between the two, and used in case of any dispute.
- **B-3.2** Tests for the determination of all characteristics given in Table 1 shall be conducted on the composite sample.

B-4 CRITERIA FOR CONFORMITY

For determining the conformity of the lot to this standard all test results on the composite sample shall meet the corresponding requirements given in Table 1.

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Electroplating Chemicals and Photographic Materials Sectional Committee, CHD 05

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Bhabha Atomic Research Centre, Hyderabad SHRI M. RAMANAMURTHY

Bharat Electronics Limited, Bengaluru SHRIMATI PADMA SHARMA

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Metal Finishers Association of India, Mumbai

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Ministry of Information and Broadcasting, New Delhi

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[REPRESENTING DIRECTOR GENERAL (Ex-

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Member Secretary MS BUNTY MUDI SCIENTIST 'B'/ASSISTANT DIRECTOR (CHEMICAL), BIS

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